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Leveraging Hybrid Pixel Electron Detection Technology to Expand Electron Microscopy Observation of Material Structures at low Voltages

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Under routine scanning/transmission electron microscopy (S/TEM) voltage conditions (80 to 300 keV), a large amount of energy is absorbed by the specimen, causing electron radiation damage [1]. Such damage is defined as the alteration of a specimen due to the electron beam irradiations, and usually results in undesired changes in physical, chemical and mechanical properties of the material under study. Two primary radiation damages are knock-on damage, and ionization damage. Knock-on damage occurs when an atom is ejected from its lattice site due to an incident electron collision, resulting in a lattice vacancy, while ionization damage occurs when outer-shell electrons of an atom are ejected. Irrespective of the type of material being studied, S/TEM experiments should be designed to reduce their effects on the results.

For example, in the last decade, characterization of two-dimensional (2D) materials have been of great interest in the field of materials science. These class of materials have large potential in future applications because of their reduced dimensions and tunable properties. Observation of 2D materials in an electron microscope comes with many challenges, one of which is electron beam irradiation, and predominantly knock-on damage when the primary electron energy is higher than about 80 keV [2]. One strategy to minimize or eliminate such alterations, and to make sure the specimen is observed as close as possible to its pristine state, is to operate the electron microscope at a lower accelerating voltage. To do so, electron detectors optimized for low keV operation are required.

As shown with several examples in this work, introduction of hybrid pixel electron detection technology (recently released Gatan Stela camera) in combination with the Gatan Microscopy Suite (GMS) allow acquisition of four-dimensional STEM (4D STEM) datasets on 2D materials, with minimal damage, at low keV (< 80 keV), and high speed (>16,000 pixels/s). Figure 1 presents simulated examples of high-resolution STEM images for a MoS_2 structure. 4D STEM dataset of 156x156x70x70 pixels, with pixel size of 0.15 Å with a 30 mrad (Figure 1A) aperture semi-angle and acceleration voltages of 40 keV and 80 keV were simulated following Kirkland multi-slice code [3]. The STEM images in Figure 1B were produced by integrating the 4D STEM dataset applying annular virtual detectors shown in Figure 1A.

Another consideration when applying low acceleration voltages for S/TEM is the resolution reduction. As the resolution is proportionally limited by the electron wavelength, achieving atomic resolution with lower energy electrons (< 40 keV) is fundamentally more challenging than using more conservative energies of 80 keV or higher. To overcome this, advanced and highly-stable geometrical aberration correctors are needed in combination with a monochromatic electron beam, which in turn limits the available electron dose, and thus demands the use of an electron counting detector and a dose efficient imaging technique such as STEM-ptychography. As shown in this work, ptychography can be efficiently applied to retrieve electron scattering events and consequently reconstruct the object function by making use of the entire bright field region. Other phase retrieval methods such as differential phase contrast (DPC) can also be applied on such datasets acquired at low voltages [4] to simultaneously identify low and high atomic number species. Figure 2 presents phase retrieval images employing the single-side band (SSB) method [5] on the same dataset shown in Figure 1, at 40 keV, for electron dose of 10^2 e-/Å^2. We observe the superior performance of ptychography phase images for extremely low electron doses in comparison to traditional STEM image modalities (ADF, ABF).

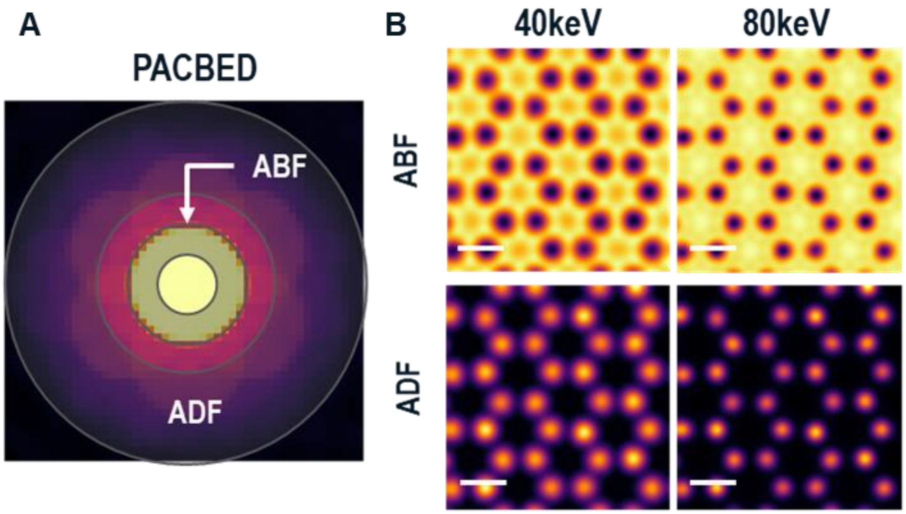

Figure 1. Figure 1. (A) Position-Averaged CBED pattern obtained by averaging out 24,336 CBED simulated patterns. A similar result is achievable by collecting 4d STEM datasets with the Stela camera. (B) ABF- and ADF-STEM images for 40keV and 80keV obtained by integrating the dataset using the virtual detectors shown in (A). Scale bars are 5 Å.

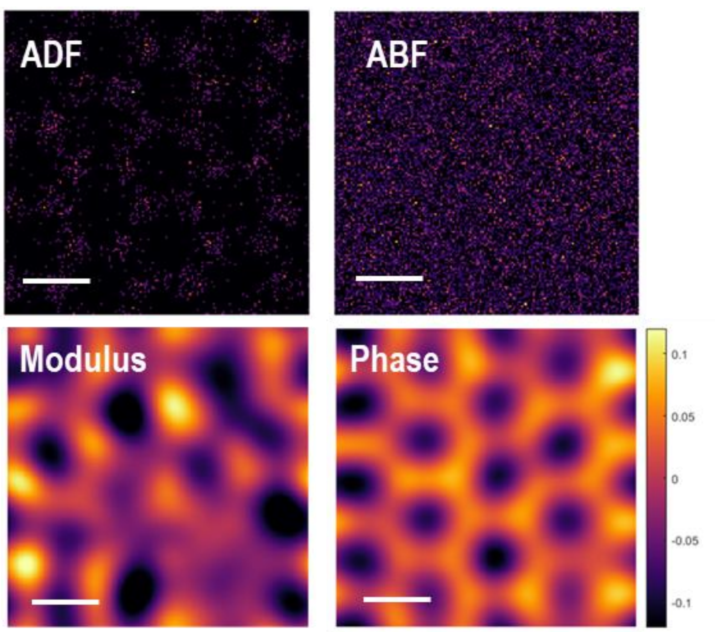

Figure 2. Figure 2. Ptychography reconstruction using SSB method for MoS2 at 40 keV applying electron dose of 102 e-/Å2 showing the superior performance of ptychography for low electron doses in comparison to traditional STEM imaging modes. Scale bars are 5 Å.

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